IN = initial condition
i = stage number
j = cycle number
M = moving phase
$N + 1$ = cold reservoir
0 = hot reservoir
PR = pure reaction
p = transfer step number
R = product
S = stationary phase
∞ = infinity

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J. F. ELY

An Empirical Correlation among Azeotropic Data

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An equation log $[10 (N_A/N_B)]$ = $-0.234\Delta t + 0.874$ serves to calculate N_A for the 251 reported alcohol–alkane azeotropes with an average ΔN_A of 0.050. The calculated value of N_A for 1108 azeotropes involving 15 different types of azeotropes has an average ΔN_A of 0.056. A modification of the equation gives an average ΔN_A of 0.028 for the alcohol–alkane azeotropes and an average ΔN_A of 0.046 for the 1108 azeotropes.

Introduction

It was early recognized that there is a relationship between the difference between the boiling points of the components of an azeotrope and its composition. Lecat (1918) used a power series to relate the composition of the azeotropes formed between members of an organic family of compounds with a single fixed component, e.g., n-alkanes and ethanol. In eq 1, x_A is the weight fraction of component A and Δt is the difference between the boiling points of the two components.

$$x_A = A_0 + A_1|\Delta t| + A_2 \overline{\Delta t^2} + A_3|\overline{\Delta t^3}| + \ldots \quad (1)$$

The necessity of using essentially all of the small number of members of a given family forming azeotropes with a fixed component in order to determine the constants in eq 1 made this of little predictive value.

In the 1940's, Mair et al. (1941) and Skolnik (1948) described graphical methods of correlating the composition of the azeotropes within an organic series, which were of limited utility. Meissner and Greenfield (1948) reported another graphical correlation which led to eq 2. These workers

$$100N_A = 55 - 0.915[T_A{}^2(T_B - T_A)] \quad (2)$$

found the graph useful for hydrocarbons (except terpenes) and halogenated hydrocarbons with alcohols, phenols, and cresols. Using 50 in place of 55 as the constant in this equation, azeotropes between halogenated hydrocarbons and ketones and aldehydes could be correlated. A decade later, Johnson and Madonis (1959) used variations of eq 2 to include a number

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of other series of compounds. Because of the many variations in the form of their equations and in the constants required, this treatment suffered from the limitations of Lecat's method.

A graphical correlation for azeotropes involving several series of compounds was proposed by Seymour (1946) in which Δt was plotted against N_A. These curves were fitted quite closely to eq 3.

$$N_A = 0.5 \cdot \frac{1}{\alpha} \text{arcsinh} \frac{\Delta t + \beta}{\gamma} \quad (3)$$

This type of equation gave useful correlations but because of the variety of constants required, it was limited in the same way as the treatments of Lecat (1918) and Johnson and Madonis (1959).

In an effort to find a more useful correlation, the data for the azeotropes between 1-alkanols and n-alkanes from Horsley's (1947) collection were combined with some data from this laboratory and the values of Δt were plotted against a variety of functions of different properties of the constituents of the azeotropes. The most useful results were obtained by plotting Δt against log N_A/N_B. The curves obtained were largely linear with slight sigmoid curvature at each end. The data for the azeotropes formed from the first three 1-alkanols and n-alkanes available at that time are plotted in Figure 1. The lines drawn through the points were calculated from the data for each alcohol by the method of least squares and fit eq 4. It is observed in Figure 1 that there is a rather systematic

$$\log \left(10 \frac{N_A}{N_B}\right) = m\Delta t + b \quad (4)$$

shift in the slope of the lines as the chain length of the alcohol is increased. This suggested that a variable parameter related

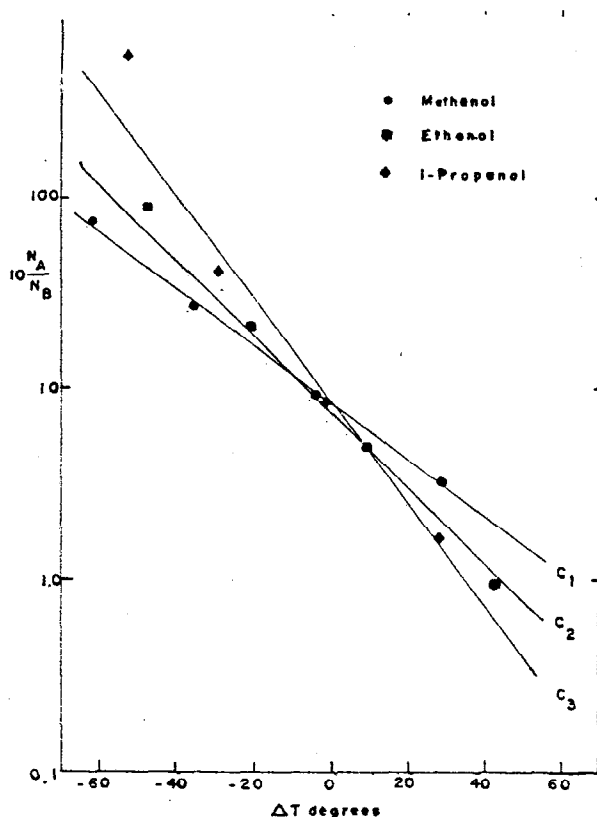Figure 1. 1-Alkanols and n-alkanes from Horsley (1947). Lines from eq 4.

Figure 2. 1-Alkanols and n-alkanes from Horsley (1947). Lines from eq 6.

to some varying property of the components might be found to adjust for this shift in slope to yield a more general, or master, equation fitting a variety of compounds. An extensive search for such a parameter led to a term we shall call ρ defined by eq 5. The values of ρ were first obtained by

$$\rho = (\text{bp (K) of a compound})/(\text{bp (K) of a hypothetical}$$
$$n\text{-alkane of the same molecular weight})\quad (5)$$

the use of a graph in which the boiling points of the n-alkanes, in degrees Kelvin, were plotted against their molecular weights. The boiling point of a hypothetical n-alkane of any desired molecular weight was read from the graph and the ρ value calculated. Later a program was prepared and the value of desired ρ's calculated by machine. The ratio ρ may be considered as one measure of the difference between the attractive forces between the molecules of a given compound and those forces between an n-alkane of the same molecular weight and, therefore, related to the nonideality of the solution of these substances. The utility of this parameter is seen by comparing Figure 1 with Figure 2. In Figure 2 the data for the same three series of azeotropes are used but instead of Δt a quantity $f(\rho)\Delta t$ is plotted against $\log (10 (N_A/N_B))$ where $f(\rho) = \rho_A/\rho_B$. When component B is an n-alkane $\rho_B = 1$, by definition, and $f(\rho) = \rho_A$. The lines in Figure 2 are represented by eq 6.

$$\log \left(10 \frac{N_A}{N_B}\right) = mf(\rho)\Delta t + b \quad (6)$$

In order to be able to test the utility of a more general master equation derived from the data for azeotropes of 1-alkanols and n-alkanes in correlating the data from other series of azeotropes, a program was begun to prepare all possible unreported azeotropes between 1-alkanols and n-alkanes which exist at a pressure of 1 atm.
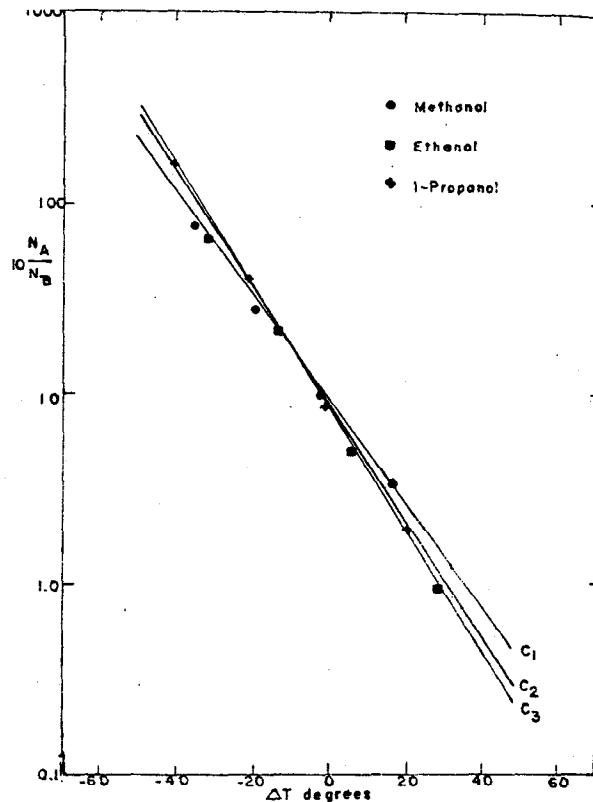
Experimental Section

Method of Preparation. The composition of the azeotropes reported in this paper were all determined by the distillation method. The approximate composition of the desired azeotrope was calculated. At first eq 4 was used and later eq 6. The still pot was charged with a mixture of the calculated composition and the still was operated at total reflux until a constant temperature was attained. A small sample, ca. 1 mL, was slowly withdrawn and the still returned to total reflux. This process was repeated until a minimum temperature was attained. Then a sample was slowly withdrawn and analyzed. This process of operating at total reflux, sample withdrawal, and analysis was continued until the distillation temperature and analysis became constant.

Materials. Over the 30 years this study continued, the quality of the materials available improved greatly. At first all materials were distilled at least twice collecting a center cut. The final cut used for preparation of azeotropes distilled over a range of not more than 0.2 °C and frequently less. All the alcohols used were subjected to this treatment but in the latter part of the work, some 99 mol % hydrocarbons from Phillips Petroleum Co. were used as received.

Distillation Equipment. The first distillation data were collected with the use of a Todd still from Todd Scientific Co. Later two vacuum jacketed columns were used. Column A has a diameter of 20 mm and is packed with ⅜-in. stainless helices for 140 cm. Column B has a diameter of 10 mm and is packed for 75 cm with ⅛-in. stainless helices. Both columns are equipped with heads which permitted control of the reflux ratio and automatic take off when desired. Reflux ratios of 50 to 1 and greater were used. Column A was used largely for purification of the component liquids. When The Petroleum Research Fund grant became available, a spinning band column, GE 196, was obtained from Precision Distillation Apparatus Co.

The early distillations were made at atmospheric pressure

Table I. Data on Azeotropes Prepared

A. Azeotropes Unreported When Prepared

| Components | Boiling points, °C | | N_A (obsd) | N_A (calcd) | ΔN_A |
	Alcohol	Hydrocarbon			
1-Butanol	117.4				
2,2,4-Trimethylpentane		99.2	0.240	0.226	0.014
2,2,5-Trimethylhexane		124.0	0.526	0.533	0.007
n-Decane		174.0	0.975	0.954	0.021
2-Methyl-2-propanol	82.4				
2,2,4-Trimethylpentane		99.2	0.682	0.688	0.006
1-Pentanol	137.5				
Heptane		98.0[a]	0.063	0.063	0.000
Octane		125.3[a]	0.299	0.269	0.030
o-Xylene		144.4	0.575	0.536	0.039
Nonane		149.4[a]	0.658	0.640	0.018
Decane		174.0[a]	0.880	0.875	0.005
3-Methyl-1-butanol	130.2				
o-Xylene		144.4	0.700	0.610	0.090
Nonane		150.8	0.71	0.74	0.03
Cyclopentanol	140.6[a]				
Octane		125.6[a]	0.289	0.234	0.055
Ethylcyclohexane		132.0[a]	0.344	0.325	0.019
Nonane		150.6[a]	0.614	0.580	0.034
Cumene		152.1[a]	0.690	0.613	0.077
1-Hexanol	156.9				
Octane		125.3	0.105	0.099	0.006
Ethylbenzene		136.2	0.074	0.165	0.091
Nonane		150.8	0.379	0.333	0.046
Decane		174.1	0.746	0.704	0.042
4-Methyl-2-pentanol	131.7				
Octane		125.6	0.350	0.349	0.001
Nonane		150.8	0.765	0.746	0.019
3-Methyl-3-pentanol	122.4				
Heptane		98.3	0.065	0.130	0.065
Octane		125.6	0.533	0.504	0.029
Toluene		110.5	0.238	0.251	0.013
Ethylbenzene		136.2	0.026	0.218	0.012
1-Heptanol	176.2				
Nonane		150.8	0.117	0.132	0.015
Decane		174.1	0.385	0.413	0.028
Undecane		195.6	0.788	0.746	0.042
1-Octanol	193.9[a]				
Nonane		149.4[a]	0.025	0.040	0.015
Decane		174.1[a]	0.21	0.17	0.04
Undecane		195.6[a]	0.52	0.48	0.04

B. Azeotropes Prepared to Check Literature Results

| Azeotropes | Boiling points, °C | | N_A (obsd) | N_A (calcd) | ΔN_A |
	Alcohol	Hydrocarbon			
1-Propanol and hexane					
Literature[b]	97.2	68.95	0.058	0.146	0.088
This research	97.2[a]	68.18[a]	0.141	0.146	0.005
2-Propanol and 2,3-dimethylbutane					
Literature	82.45	58.0	0.124	0.173	0.049
This research	82.3[a]	58.0[a]	0.179	0.173	0.006
3-Methyl-1-butanol and o-xylene					
Literature	131.9	142.6	0.570	0.674	0.104
This research	131.8[a]	143.9[a]	0.700	0.674	0.026
3-Methyl-1-butanol and cumene					
Literature	131.9	152.8	0.955	0.774	0.181
This research	131.5[a]	152.7[a]	0.831	0.774	0.057

[a] These data were measured at atmospheric pressure and corrected to 760 Torr. All other reported data were measured at 760 Torr.
[b] All literature data, unless otherwise noted, are from Horsley (1973).

which varied between 735 and 745 Torr. Later, the stills were operated at 760 Torr under a nitrogen atmosphere. The pressure was controlled with a Cartesian Manostat from Emil Greiner Co., G15080 Model 5, and measured with an Absolute Manometer, Greiner No. 41383.

Methods of Analysis. The first analyses were made by density determinations with calibrated pycnometers. Calibration curves were prepared from a series of standard samples bracketing the composition of the given azeotrope. Other analyses were made with a Bausch and Lomb Refractometer

Table II. Selected Values for ρ

Hydroxy compounds		Hydrocarbons	
Water	2.65	Benzene	1.09
Methanol	1.77	1,3-Cyclohexadiene	1.08
1-Propanol	1.33	Cyclopentane	1.06
1-Heptanol	1.11	Methylcyclopentane	1.02
2-Propanol	1.28	2-Methylbutane	0.973
2-Methyl-2-propanol	1.13	2,2,4-Trimethylpentane	0.935
Cyclopentanol	1.21		

Table III. Slope and Intercept Constants for 1-Alkanol n-Alkane Series

	m	b
[A. For Eq 4]		
Methanol series	−0.0151	0.932
Ethanol series	−0.0207	0.869
Propanol series	−0.0265	0.936
Butanol series	−0.0249	0.862
Pentanol series	−0.0270	0.935
Hexanol series	−0.0291	0.992
Heptanol series	−0.0321	0.913
Octanol series	−0.0349	1.027
All 1-Alkanols	−0.0234	0.874
[B. For Eq 6]		
Methanol series	−0.0267	0.933
Ethanol series	−0.0306	0.867
Propanol series	−0.0316	0.939
Butanol series	−0.0310	0.862
Pentanol series	−0.0319	0.936
Hexanol series	−0.0331	0.985
Heptanol series	−0.0357	0.913
Octanol series	−0.0384	1.027
All 1-Alkanols	−0.0317	0.906

No. 33-45-58 using calibration curves prepared as above. Finally, analyses were made with an Aerograph Chromatograph No. 200. In many cases analyses were made by two of the methods.

Discussion

The first tests of the effectiveness of the ρ factor in improving the correlation between N_A and Δt were made graphically. The function $\Delta t f(\rho)$ was plotted against $10\,(N_A/N_B)$ on semilogarithmic paper. When a 1130 IBM computer became available, values for m and b in eq 6 were calculated by the least-squares method and a few tests were made on azeotropes of other alcohols and hydrocarbons. In 1970 this department obtained a Hewlett-Packard 9810 A calculator and all calculations on correlations reported in this paper were made on this machine.

Fourteen new azeotropes between 1-alkanols and n-alkanes have been prepared. Attempts to form azeotropes with 1-nonanol and 1-decanol at atmospheric pressure resulted in the dehydration of the alcohols. In Table I all azeotropes prepared in this laboratory are reported. Part A lists those not reported in the literature at the time they were prepared. In addition to the 14 mentioned above, azeotropes were prepared involving types of alcohols and hydrocarbons not widely represented in the literature. In part B data are given for four azeotropes whose literature data gave poor correlation with our master equation. The significance of N_A calculated and ΔN_A will be discussed later.

Representative values of ρ are given in Table II to illustrate the variation of this parameter with different types of compounds. The values for m and b for eq 6 were calculated for each series of azeotropes formed from a single 1-alkanol with

n-alkanes. Finally, m and b for the composite curve for all 31 azeotropes formed from 1-alkanols and n-alkanes were calculated. These values are given in Table III. In accord with the change in slope of the lines in Figure 2, there is an increase in the value of m as the length of the alcohol chain increases. No explanation has been found for the fact that the value of m for 1-propanol is out of line. No regularity has been observed for the variation of b.

Substitution of the m and b values calculated from the 31 1-alkanol–n-alkane azeotropes into eq 6 gives eq 7. This equation is used as a master equation to correlate the data from other series of azeotropes.

$$\log\left(10\,\frac{N_A}{N_B}\right) = -0.0317 f(\rho)\Delta t + 0.906 \tag{7}$$

Similarly eq 8 is obtained from eq 4. Equation 7 was used

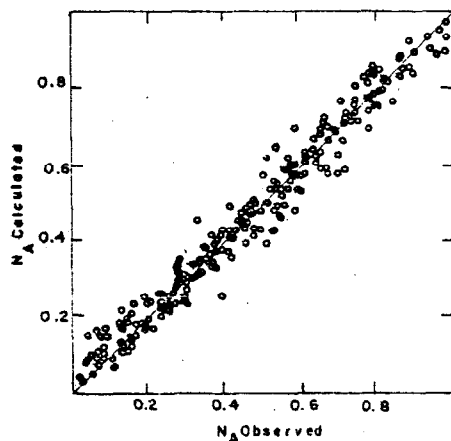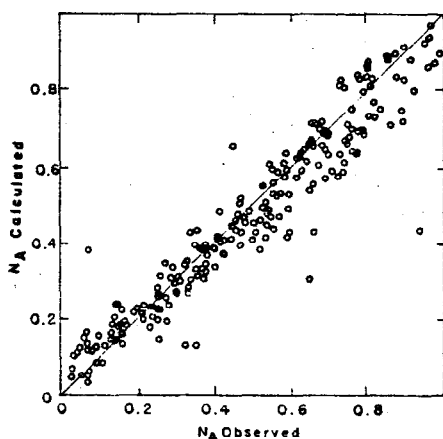$$\log\left(10\,\frac{N_A}{N_B}\right) = -0.0234\Delta t + 0.874 \tag{8}$$

Table IV. Summary of Differences between Observed and Calculated Composition of Azeotropes

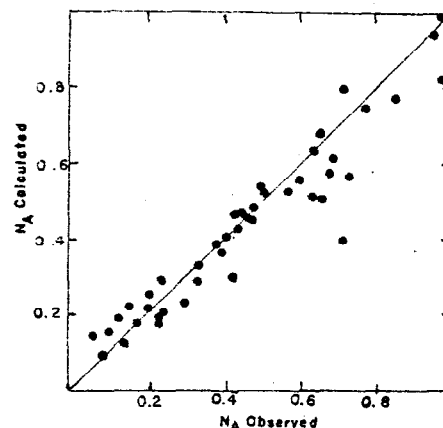|	Type of azeotrope	No. of items	Difference between Observed and Calculated N_A		
			Eq 7, Av ΔN_A	Eq 8, Av ΔN_A	Eq 2, Av ΔN_A
1.	n-Alkanes and 1-alkanols	31	0.019	0.037	0.111
2.	All[a] hydrocarbons and alcohols	220	0.028	0.053	0.038[b]
3.	Hydrocarbons and alkoxyalcohols	68	0.050	0.089	0.074[b]
4.	Monohalides and alcohols	144	0.048	0.030[c]	0.053
5.	Hydrocarbons and ketones	45	0.051	0.070	0.068[b]
6.	Hydrocarbons and carboxylic acids	76	0.060	0.081	0.075[b]
7.	Hydrocarbons and esters	168	0.060	0.067	0.127
8.	Hydrocarbons and nitriles	24	0.066	0.067	0.127
9.	Organic sulfides and alcohols	17	0.059	0.072	0.065[b]
10.	Hydrocarbons and water	27	0.046	0.071	e
11.	Ethers and water[d]	98	0.054	0.051[c]	e
12.	Alcohols and water[d]	31	0.036	0.009[c]	e
13.	Esters and water[d]	125	0.048	0.036[c]	e
14.	Amines and water[d]	24	0.032	0.050	e
15.	Carboxylic acids and water[d]	10	0.054	0.011[c]	e
Total		1108	0.046	0.56	—

[a] Does not include 31 items above. [b] Lower than those from eq 8. [c] Lower than those from eq 7. [d] Only those organic compounds boiling above 150 °C. [e] ΔN_A values are 0.2 and larger.

Table V. Data for Series Fitting Eq 7 Poorly

Type of azeotrope	No. of items	New values		Av ΔN_A	From eq 7: Av ΔN_A
		Constants for eq 6			
		m	b		
Nonaromatic hydro-carbons and aromatic hydrocarbons	23	−0.115	0.992	0.036	0.18
Hydrocarbons and alkyl sulfides	42	−0.086	1.01	0.044	0.15
Hydrocarbons and halides	27	−0.055	1.02	0.034	0.066
Hydrocarbons and nitriles	24	−0.036	1.10	0.038	0.066

Figure 5. Comparison of N_A (obsd) and N_A (calcd) from eq 7. Hydrocarbons and ketones, series 5, Table IV.

Figure 3. Comparison of N_A (obsd) and N_A (calcd) from eq 7. All hydrocarbons and all alcohols, series 1 and 2, Table IV.
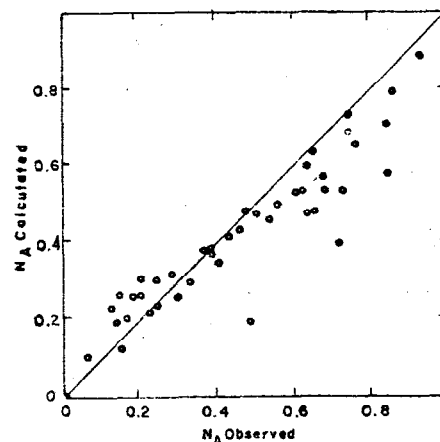
Figure 6. Comparison of N_A (obsd) and N_A (calcd) from eq 8. Hydrocarbons and ketones, series 5, Table IV.

Figure 4. Comparison of N_A (obsd) and N_A (calcd) from eq 8. All hydrocarbons and all alcohols, series 1 and 2, Table IV.
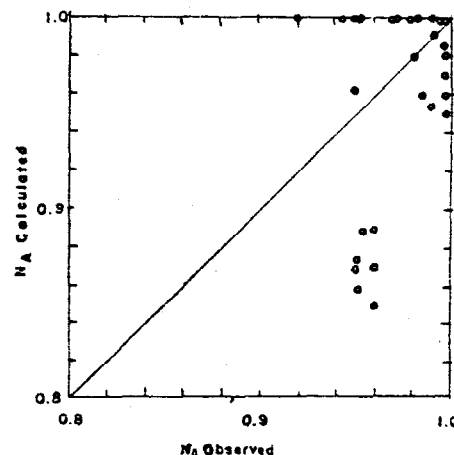
Figure 7. Comparison of N_A (obsd) and N_A (calcd) from eq 7. Esters and water, series 11, Table IV.

to obtain calculated values for N_A for over 1100 azeotropes including all prepared here and the rest from those listed by Horsley (1973). (Only data for those azeotropes for which no uncertainty was indicated in the collection of Horsley (1973) were used. Calculations were made for N_A for a limited number of azeotropes for which uncertainty was indicated and while some results agreed quite well with the observed values, most of them gave rather large differences and all were omitted.) From these data, values for ΔN_A, the absolute value of the difference between the calculated and observed N_A for each azeotrope, were calculated. In a similar manner another set of calculated values for N_A and ΔN_A were obtained from

eq 8. Finally, another set of calculated N_A and ΔN_A were obtained from eq 2 for those series which gave useful results. All of these results are given in Table IV.

In Table IV we see that eq 7 gives an average ΔN_A of 0.046 for more than 1100 azeotropes from fifteen different series of compounds. The corresponding average ΔN_A from eq 8 is 0.056, which is only slightly poorer. However, if we compare the first three series and the tenth series (almost 350 azeotropes) in which the compounds are most nearly like those
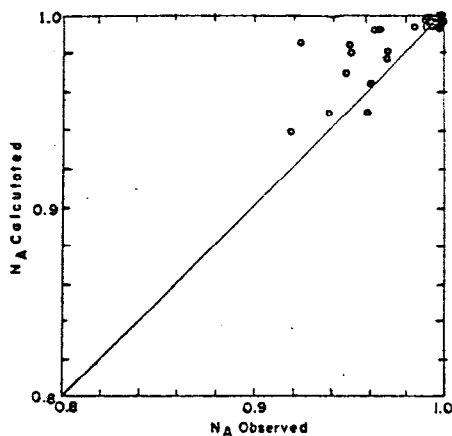
Figure 8. Comparison of N_A (obsd) and N_A (calcd) from eq 8. Ethers and water, series 11, Table IV.
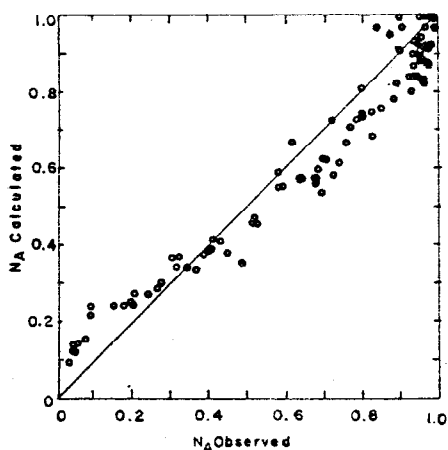

Figure 9. Comparison of N_A (obsd) and N_A (calcd) from eq 7. Alcohols and water, series 12, Table IV.
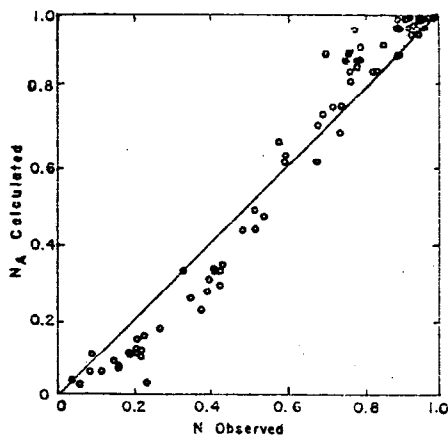

Figure 10. Comparison of N_A (obsd) and N_A (calcd) from eq 8. Alcohols and water, series 12, Table IV.

Figure 11. Distribution of ΔN_A values. N_A calculated from eq 7. All hydrocarbons and all alcohols, series 1 and 2, Table IV.
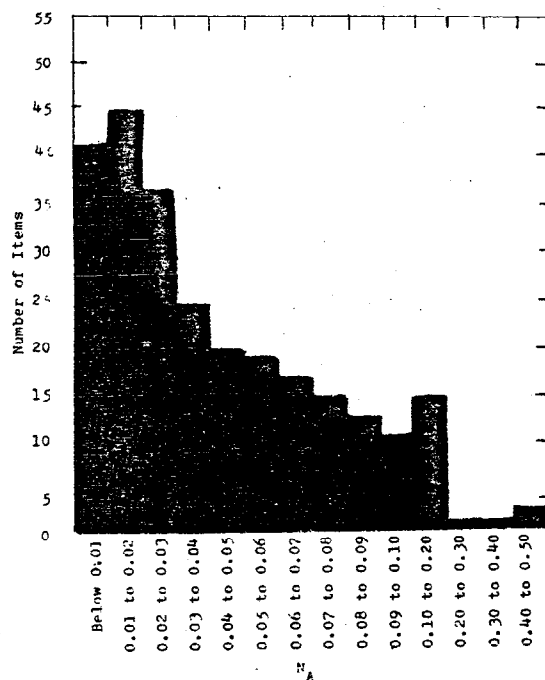

Figure 12. Distribution of ΔN_A values. N_A calculated from eq 8. All hydrocarbons and all alcohols, series 1 and 2, Table IV.

used to obtain eq 7, the superiority of eq 7 over eq 8 is much greater. Also series five, six, and nine give considerably better correlations with eq 7 than with eq 8. While eq 2 gives better results than eq 8 in five series, in no case does it give better results than eq 7.

The ΔN_A values calculated for the compounds boiling below 150 °C in series 11–14 were much higher than for the higher boiling compounds and were omitted. This may result from greater H-bonding due to greater solubility.

Aside from those involving water, the only series of azeotropes for which eq 8 gave better results than eq 7 was that of monohalides and alcohols. The presence of a halogen atom in either component of an azeotrope led to poor correlations. No useful results were obtained with haloalcohols, haloacids, nor polyhalides. Meissner and Greenfield (1948) reported poor results with terpenes. While we have found that a few azeo-
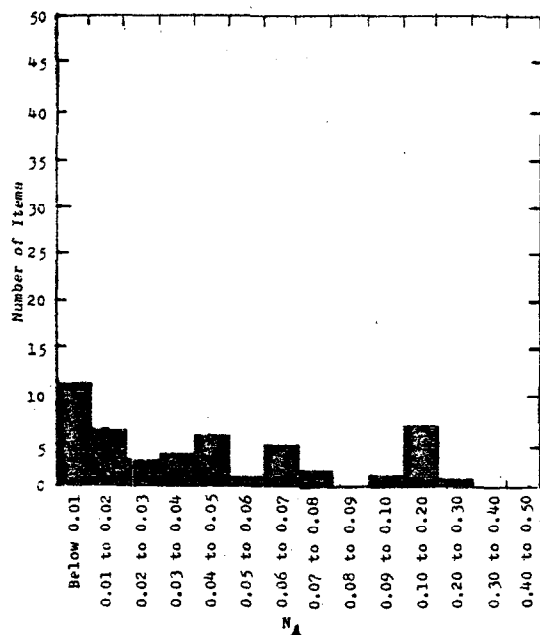
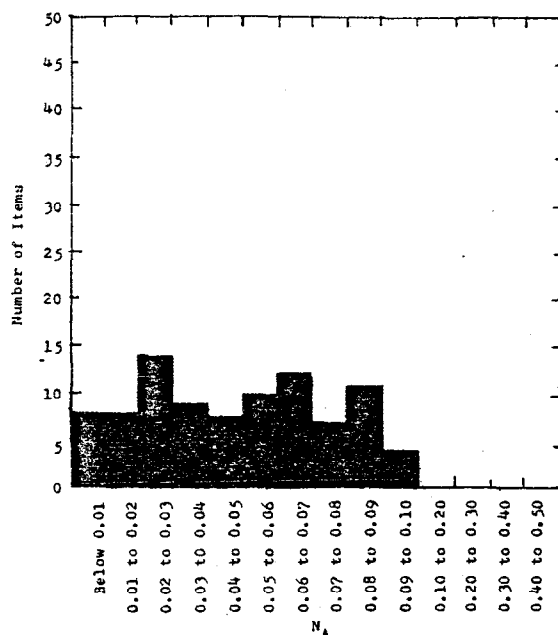**Figure 13.** Distribution of ΔN_A values. N_A calculated from eq 7. Hydrocarbons and ketones, series 5, Table IV.

Figure 15. Distribution of ΔN_A values. N_A calculated from eq 7. Ethers and water, series 11, Table IV.
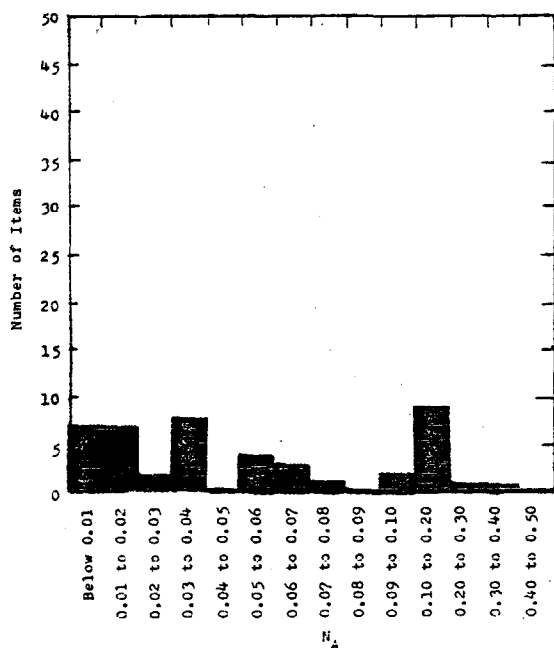

Figure 14. Distribution of ΔN_A values. N_A calculated from eq 8. Hydrocarbons and ketones, series 5, Table IV.
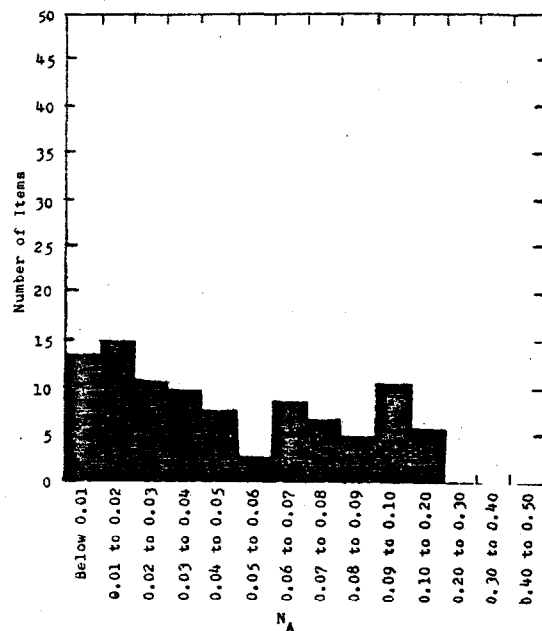

Figure 16. Distribution of ΔN_A values. N_A calculated from eq 8. Ethers and water, series 11, Table IV.

tropes involving terpenes give useful correlation, most of them do not and none have been included in this study.

It was observed earlier that in the series of azeotropes formed from n-alkanes and 1-alkanols the value of m increased with the length of the alcohol chain. This must be related to the increasing ideality of the resulting solutions. In agreement with this observation, it is found that with series of azeotropes in which the constituents are increasingly alike, the value of m increases accordingly. Data are given in Table V for m and b values, calculated from eq 6 for four series of azeotropes which gave poor correlations with eq 7.

As anticipated, the value for m for the first series is largest. The difference between the value of ΔN_A calculated from the m and b of each series and that calculated from eq 7 becomes smaller as the new m approaches that of eq 7.

A graphical comparison of the agreement between the calculated and observed values for four of the 15 series of azeotropes studied is shown by Figures 3–10. (The four series 2, 5, 11, and 12 were suggested by a referee. The data for series 1 were added to series 2 so as to include all hydrocarbon-alcohol azeotropes.) That N_A values calculated with eq 7 for the 251 alcohol–hydrocarbon azeotropes agree more closely with observed values than those calculated with eq 8 is seen by a comparison of Figures 3 and 4. The difference is less for the ketone–hydrocarbon azeotropes as seen in Figures 5 and 6. That the converse is true for those azeotropes between water and compounds capable of forming H bonds with water is observed in Figures 7 through 10. It should be noted that Figures 7 and 8 are plotted on a different scale from the others. The only series of azeotropes involving water for which eq 7
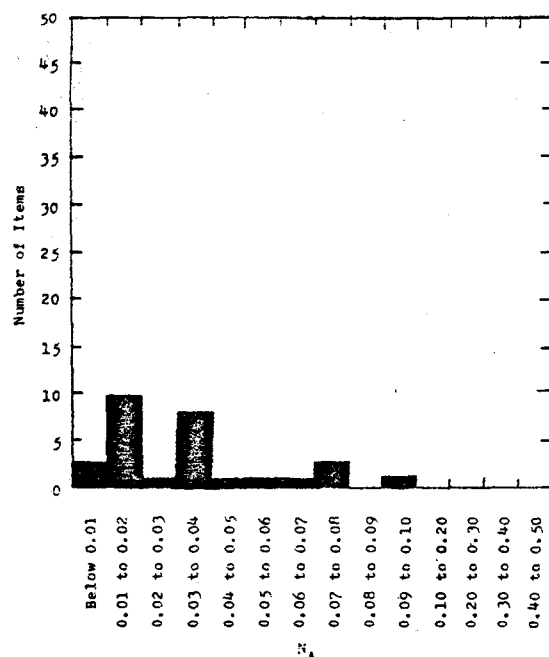
Figure 17. Distribution of ΔN_A values. N_A calculated from eq 7. Alcohols and water, series 12, Table IV.

Figure 18. Distribution of ΔN_A values. N_A calculated from eq 8. Alcohols and water, series 12, Table IV.

gives better results that eq 8 is the water–hydrocarbon series which most closely resembles the alcohol–hydrocarbon series from which eq 7 was derived.

Figures 11–18 depict the distribution of the N_A values obtained with eq 7 and eq 8. Again the superiority of eq 7 over eq 8 for the alcohol–hydrocarbon series is seen from Figures 11 and 12.

While the use of the corrective parameter ρ in eq 7 gives an equation which correlates more azeotropes and more kinds of azeotropes than any previously reported equation, it has obvious limitations. However, until that time when the desired calculations can be made from theoretical principles, it is suggested that other similar factors may be found which, perhaps combined with the ρ factor, will give equations with greater utility. All our experimental work has been conducted at atmospheric pressure. No attempt has been made to study the effect of pressure on our correlations.

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Nomenclature

$A_0, A_1, A_2, \ldots$ = constants in a power series
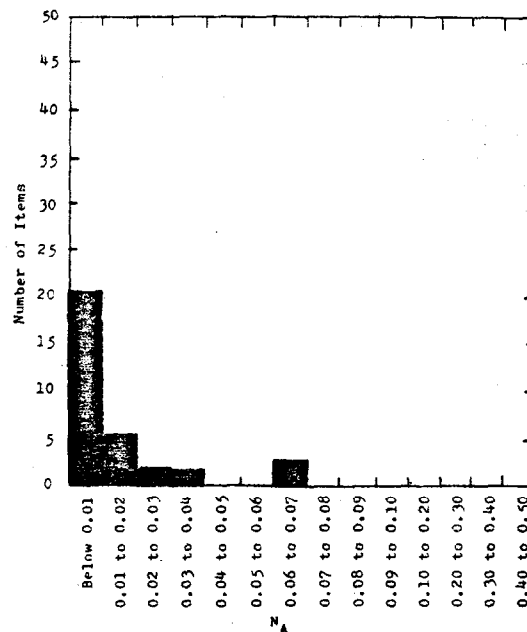b = constant (intercept in eq 4 and 6)

$\Delta N_A = |N_A(\text{obsd}) - N_A(\text{calcd})|$
$f(\rho) = \rho_A/\rho_B$
m = constant (slope in eq 4 and 6)
N_A = mole fraction of component A
N_B = mole fraction of component B
ρ = a defined ratio (see eq 5)
T = boiling point temperature, K

Greek Letters

α = arbitrary constant
β = arbitrary constant
γ = arbitrary constant

Subscripts

A = more polar component
B = less polar component

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